



Processing Hypalon[®]

This technical information presents some guidelines for consistent, satisfactory processing of compounds of Hypalon[®] synthetic rubber. Processing Hypalon[®] in a rubber products plant may be defined as the entire series of operations beginning with the arrival of the raw material on the receiving dock and continuing until the finished product is in the warehouse ready for shipment. Since most dry compounding and processing is done with Hypalon[®] 40, or one of its viscosity variants, the bulk of the discussion is based on these materials.

With the exception of the coating grades, all types of Hypalon[®] can be mixed in internal mixers and, for highly loaded compounds, this is the only practical mixing method. Most compounds are one-pass mixed using the 'upside-down' technique. When the mix is complete, the batch should be dropped on a sheet-off mill, quickly cooled, and stored in a cool, dry environment.

A wide variety of compounds are extruded using both cold and warm feed equipment. Cold feed, long barrel extruders take compounded strip at ambient temperatures, whereas warm feed, short barrel extruders are fed strips of stock previously warmed on a mill.

Large volumes of compounded Hypalon[®] are calendered into sheets for pond or landfill liners and single-ply roofing. Hypalon[®] 45 is particularly suitable for liners and roofing, while the general purpose polymers are preferred for other calendered applications.

Compounding practices for compression, injection, and transfer molding are all similar, but the cure system, acid acceptor, and process aid packages should be carefully selected based on the properties required and important machine/mold surface considerations. For good transfer molding, it may be necessary to prewarm the preforms. For reciprocating screw injection molding presses, it is desirable to use a lower viscosity compound to give the best feed and inject properties. The cure rate and machine conditions must be balanced for stock scorch versus complete mold filling.

Sulfur and maleimide cured compounds of Hypalon[®] can be vulcanized in steam autoclaves, CV steam tunnels, air ovens, microwaves, and LCM equipment. Fast cures are desired for continuous curing operations to get maximum throughput. When there is no pressure on the extrudate during cure, a high viscosity polymer, an effective desiccant, and a vacuum extruder should be used to eliminate porosity. Peroxide cured compounds can be cured in steam autoclaves (if all the air is purged before curing starts) or CV steam tunnels, but peroxide compounds are not used in air or microwave ovens because of oxygen interference.

The manner in which processing of Hypalon[®] 45 and Hypalon[®] 48 differs from that of Hypalon[®] 40 is reviewed in a section at the end of the bulletin. Hypalon[®] 20 and Hypalon[®] 30 are normally processed as solvent coatings, and are discussed in other reports.

Handling Precautions

Safe Handling

Hypalon[®] synthetic rubber contains low levels of carbon tetrachloride (CCl₄) and chloroform (CHCl₃) as residues from manufacturing. Carbon tetrachloride and chloroform are classified by The International Agency for Research on Cancer (IARC), the National Toxicology Program (NTP), and the American Conference of Governmental Industrial Hygienists (ACGIH) as substances for which there is limited evidence of carcinogenicity to humans. Under the Occupational Safety and Health Act (OSHA), exposures to carbon tetrachloride must be kept below the permissible exposure limit of 10 ppm and chloroform must be kept below the permissible exposure limit of 50 ppm. When large quantities of raw polymer are stored or processed in enclosed areas with restricted air exchange or ventilation, the air must be monitored for carbon tetrachloride and chloroform; and, if necessary, ventilation must be supplied to comply with OSHA regulations.

For additional information concerning this and other potential industrial health hazards when handling Hypalon[®], see DuPont Dow Elastomers technical bulletin "Toxicity and Handling Guide," and The Material Safety Data Sheet (MSDS).

Compounding Ingredients

Compounding ingredients, including peroxides and lead-based curatives, used with Hypalon[®] to prepare finished products, may present hazards in handling and use. Before proceeding with any compounding or processing work, consult and follow label directions and handling precautions from suppliers of all ingredients.

Waste Disposal

All grades of Hypalon[®] raw polymer exceed the regulated maximum leachate level for carbon tetrachloride in the U.S. Environmental Protection Agency (EPA) toxicity characteristic leaching procedure (TCLP test); therefore, discarded polymer should be considered a Resource Conservation and Recovery Act (RCRA) hazardous waste by anyone who generates more than 220 lb of total hazardous waste (includes waste from all sources) at a site in a calendar month.

The level of residual carbon tetrachloride in compounds is less than in Hypalon[®] because of dilution by other ingredients in the compound. Compounding ingredients, processes, and applications vary significantly, so you will need to reach your own conclusions as to whether waste generated by your process exceeds TCLP regulatory limits subject to RCRA regulations.

Hypalon[®] contains no lead, but lead containing materials are sometimes compounded with it. If lead can be extracted from the compound in amounts that exceed the regulatory limit of EPA's TCLP test, then the compounded waste is a RCRA hazardous waste (Federal Register, 3/29/90, p11862; 40CFR 261.24), and must be handled as such. The amount of lead extracted in the TCLP test will depend on the amount and type of lead compound present, other compounding ingredients, and compounding and/or curing conditions. If you have any questions, please contact your DuPont Dow Elastomers sales representative.

General Characteristics of Hypalon[®]

Hypalon[®] is the trademark assigned to a group of sulfur and peroxide curable elastomers that are based on chlorosulfonated and chlorinated polyethylene. These polymers are manufactured in such a manner as

to produce an elastomer with a completely saturated backbone and pendant groups suitable for varied approaches to vulcanization. As a result of this configuration, vulcanizates of Hypalon[®] synthetic rubber are extremely resistant to attack by ozone, oxygen and weather. In addition, properly prepared vulcanizates of Hypalon[®] are outstanding in resistance to deterioration by heat, oils, and many chemicals and fluids. However, there are chemical and physical differences between the various types which affect processibility and properties. The compounder must select the type that will best produce the desired end product within the constraints of each unique manufacturing situation.

The principle features of each type are summarized in *Figure 1*.

Distinguishing Polymer Processing Characteristics

Thermoplasticity

Uncured Hypalon[®] synthetic rubber is more thermoplastic than other commonly used elastomers. It is generally tougher at room temperature, but softens more rapidly as temperature is increased by working on a mill or in an internal mixer. Viscosity temperature relationships for Hypalon[®] 40 and SBR are shown in *Figure 2*.

The low viscosity of Hypalon[®] 40 at elevated temperatures permits extrusions that are low in die swell, smooth at fast rates, and sharply defined. This softness under slightly warmed conditions (40°C), makes building operations practical, giving good ply adhesion and knitting without the use of tackifiers. However, because of this same softness, immediate cooling of extrusions is necessary to prevent distortion. Also, variations in stock temperature may cause variable extrusion rates and calendared sheet with uneven gauge and a rough surface.

Processing of either very soft or very stiff compounds can be improved by using one of the other grades of Hypalon[®]. The high viscosities of Hypalon[®] 4085, and HPG-6525 help eliminate trapped air and blistering during processing and curing of highly extended or low durometer stocks. They also help minimize distortion of these compounds after extrusion and during open steam curing. The low viscosity of 40S permits easier processing of stocks containing highly reinforcing fillers or small amounts of plasticizers.

Resistance to Breakdown

Hypalon® does not break down in the sense that natural rubber and sulfur-modified Neoprene types do. These rubbers undergo an irreversible chemical change (a reduction in molecular weight) when they are worked. The reduction of toughness and boardiness that occurs as Hypalon® is milled is strictly temperature related, i.e., temporary and reversible. This means that compounds of Hypalon® can be reworked without significant change in processing behavior, as long as scorch is not a factor. It also means that processing operations should be consistent from batch-to-batch, if constant temperatures are maintained.

Because of thermoplasticity and lack of breakdown, compounds based on Hypalon® tend to be dry and non tacky at *room temperature*, but *warmed* stock gives good flow, knitting, and ply adhesion.

Typical viscosity relationships for the general purpose types, Hypalon® 4085, 40, and 40S, are shown in *Figure 3*, along with viscosity temperature curves for Hypalon® 45 and 48. Note that Hypalon® 45 and 48 are more temperature sensitive than any of the general purpose Hypalon® 40 types.

Figure 1. Selection Guide for Hypalon®

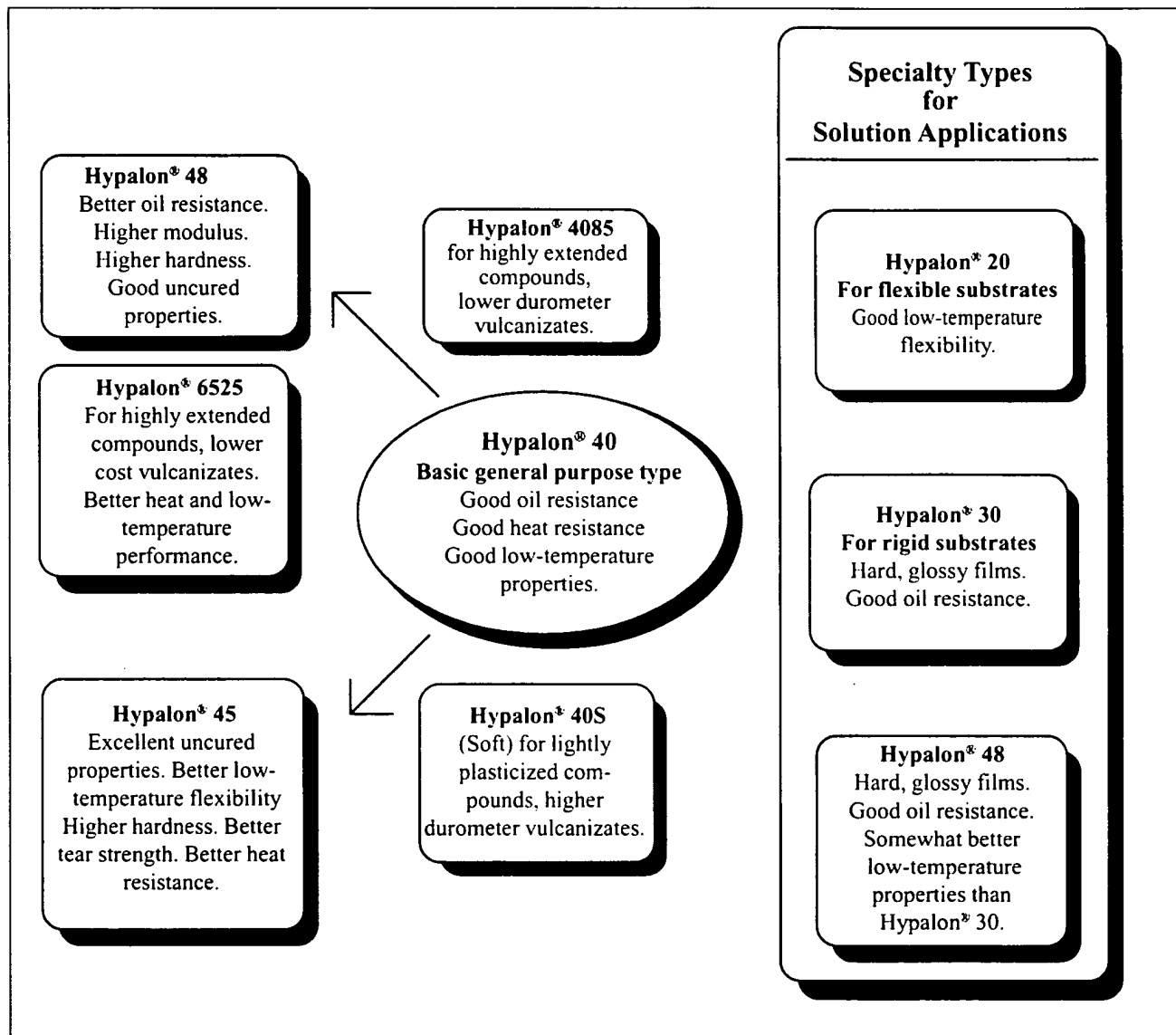


Figure 2. Viscosity/Temperature Relationship of Hypalon® 40 and SBR1500

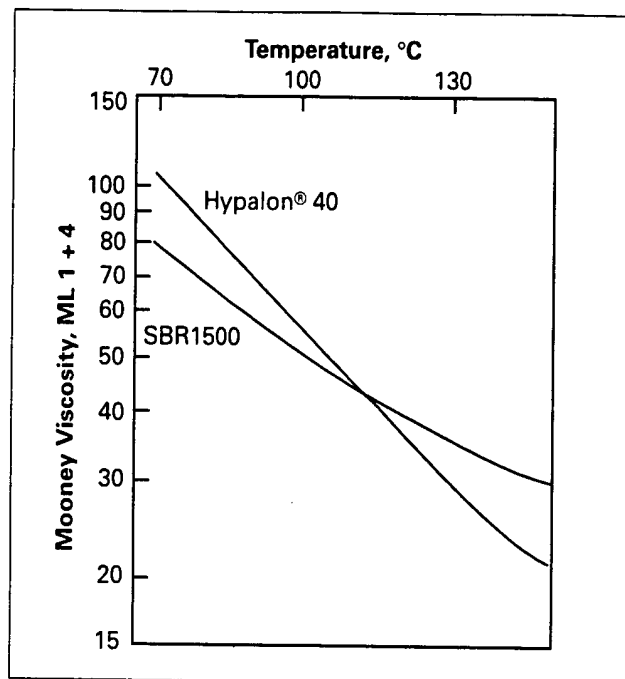
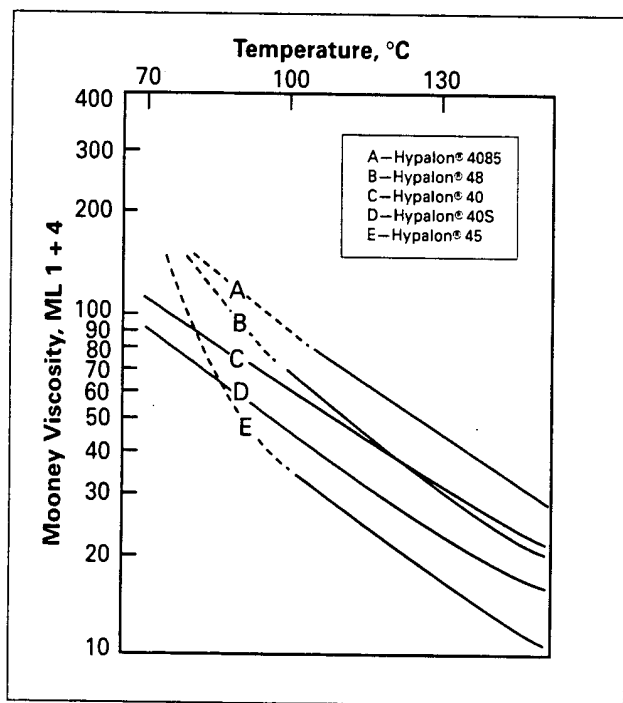


Figure 3. Viscosity/Temperature Relationship of Hypalon Polymers



Effects of Compounding on Processing

The following paragraphs deal with those aspects of compound design which affect the processability of Hypalon®. Compounding for specific performance characteristics is discussed more completely in other technical bulletins on Hypalon.

Raw Polymer Selection and Storage

The plasticity or viscosity of compounds of Hypalon® can be controlled, in part, by selecting the right viscosity grade. Hypalon® 40S is used to reduce the viscosity of stiff compounds that have low amounts of plasticizer or contain high structure carbon black. Hypalon® 4085 adds uncured strength to soft compounds that contain large quantities of plasticizers or compounds that are highly extended.

The viscosity, processing characteristics and cure rate of raw Hypalon® do not change appreciably under typical storage conditions. Temperature is a factor, however, because of thermoplasticity. High storage temperatures promote "cold flow" of the chips in a bag and, given sufficient pressure and time, this can lead to massing of the chips and even rupture of bags. For this reason, storage away from heat sources and first-in/first-out rotation of polymer inventory are recommended.

Zinc-Containing Materials

Zinc bearing additives should *never* be used with compounds based solely on Hypalon®, and their harmful effects deserve special mention here because they are commonly found in rubber processing areas. Zinc compounds (e.g., zinc oxide and zinc stearate) seriously reduce heat resistance (over 120°C) of vulcanizates of Hypalon® even when present in small amounts as accidental contaminants. Since heat resistance is often a major reason for using Hypalon® synthetic rubber in a given product, even the sparing use of zinc compounds as dusting agents on slabs or extrudates should be avoided. If zinc oxide is used to cure a diene elastomer (SBR, NBR) *blended with* Hypalon®, the heat resistance of the blend at 120°C will be less than that of Hypalon® alone.

Processing Aids

All practical compounds based on Hypalon® contain release agents and other processing aids. Types which have been used satisfactorily are discussed below. Some types function better at cool to moderate temperatures, others function better at higher temperatures. It is good practice to use small amounts of several aids to insure release over a range of temperatures without exceeding the limits of compatibility of any of the individual materials with Hypalon®.

Microcrystalline waxes, petrolatum and paraffinic waxes are effective, but they must be used sparingly; if used in excess, they bloom. Victory® Amber wax, Multiwax® 180M, and Vanwax® H have been used satisfactorily.

Polyethylene glycol is very effective at low processing temperatures. Carbowax® 3350 has been used quite frequently.

Low molecular weight polyethylenes are effective at higher processing temperatures. A-C Polyethylene 617A and A-C Polyethylene 1702, DFDA-0053, and Epolene® N-14 have been used satisfactorily. Low molecular weight polyethylene is rarely used as the only process aid, since it is not particularly effective below its fluxing temperature. When added on a cold mill, it can actually aggravate sticking. Proprietary polymer blends, such as Vanfre® HYP, improve flow and mold release.

Stearic acid, stearates (*other than zinc*) and many proprietary process aids are safe and effective with magnesia cures. They should *not* be used with litharge cures as they can seriously reduce scorch safety. For a more complete discussion of process aids, see DuPont Dow Elastomers technical bulletin "Selecting a Plasticizer and Processing Aid."

Plasticizers

Large amounts of aromatic oils tend to make compounds of Hypalon® sticky. Blends of naphthenic and aromatic plasticizers give easier processing compounds than aromatic oils alone. Naphthenic oils are compatible in amounts up to 15–20 phr. Reinforcing fillers (e.g. silica or N550 carbon black) are used to increase the green strength of highly plasticized compounds. Such stocks are sometimes soft and tender when hot, even though they may not be sticky.

Factice is compatible with Hypalon® and helps dry up stickiness caused by large amounts of aromatic oil. It also gives soft compounds more body and makes them easier to handle, but with some loss in heat and compression set resistance and color stability. For more detailed information on plasticizers, consult DuPont Dow Elastomers technical bulletin "Selecting a Plasticizer and Processing Aid."

Mixing

Production compounds of Hypalon® synthetic rubber are mixed in internal mixers and on mills. Internal mixers are preferred for speed and processing safety, but mill mixing is used satisfactorily for small batches, or where the particular factory situation warrants it. Important considerations in mixing Hypalon® are reviewed in the following discussion.

Internal Mixers

Batch Size and Loading Factor

Efficient internal mixing with Hypalon® requires a higher loading factor than with most other polymers; 70 to 75% of the volume of the mixing chamber is desirable. Batch sizes are calculated as follows:

$$\text{Batch size in kg or lb} = \frac{\text{Mixer capacity in kg or lb of water}}{\text{loading factor} \times \text{specific gravity}}$$

The capacities of various internal mixers are shown in *Table I*, along with batch size for these same mixers based on a specific gravity of 1.0, and a loading factor of 0.7. This load factor is satisfactory for many compounds of Hypalon®. Multiply these batch sizes by the specific gravity of your compound to arrive at your batch size. Some compounds are quite tolerant of batch size variations, but with others the determination of optimum batch size is essential to efficient mixes with good dispersion.

The loading factor defines that portion of the total mixer capacity actually occupied by the mixed batch. Loading factor is dependent upon several variables including:

Condition of the Mixer—Wear on the rotor and chamber increases the capacity of the mixer and requires a higher theoretical loading factor.

Stock Viscosity—(often correlates with cured hardness) Stiff compounds, those containing little plasticizer, those based on high viscosity polymers, and compounds containing reinforcing fillers tend to generate excessive heat. Lower loading factors may be necessary to control temperature.

Rheological Properties of the Polymer—With polymers that soften thermally, such as Hypalon®, loading factor may be increased, which will allow a larger batch size.

Cooling Efficiency and Available Power—Loading factor may have to be reduced to control stock temperature when mixer cooling capabilities are marginal, or to stay within the power limits of the equipment.

Table 1
Typical Batch Sizes for Hypalon® in Internal Mixers

Basis: Loading Factor—0.7: Compound Specific Gravity—1.0

Adamson Intermix (Adamson Division) Wean United, Inc.

Mixer Size	K-2A	K-4	K-5	K-6	K-7
Net Volume of Chamber, liter	46.7	68.8	112	160	266
Chamber Capacity, kg water	46.7	68.6	112	160	266
Typical Batch Size, kg	32.6	48	78	112.5	186

Bolling Spiral-Flow Intensive Mixer (Stewart Bolling & Co., Div. of Intercote Automation, Inc.)

Mixer Size	No. 3	No. 4	No. 10	No. 12
Net Volume of Chamber, liter	56.7	83.5	200	262
Chamber Capacity, kg water	56.7	83.5	200	262
Typical Batch Size, kg	40	58.5	139.7	183

Farrel-Banbury Mixers (Farrel Connecticut Division, USM Corp)

Mixer Size	3A	9	11	27
Net Volume of Chamber, liter	70.8	187	245	618
Chamber Capacity, kg water	70.8	187	245	618
Typical Batch Size, kg	49.4	131	171.5	432

Werner & Pfleider Internal Mixer (Werner & Pfleider Corp.)

Mixer Size	GK 50UK	GK 100UK	GK 160UK	GK 230UK
Net Volume of Chamber, liter	86	156	255	330
Chamber Capacity, kg water	86	156	255	330
Typical Batch Size, kg	60	109	178	231

The loading factor must be adjusted as experience is gained with a particular compound on specific mixing equipment. Where a compound may be mixed in more than one mixer, each mixer *may* require a different batch size (load factor). Overloading the mixer may result in a hot and perhaps incomplete mix. Underloading gives lower temperature, but longer mix cycles with the risk of poor dispersion.

Mixing Procedures

Upside-down loading gives the most efficient mix with most compounds of Hypalon®. Fillers are charged first, followed by the metal oxide, process aids, plasticizers and the polymer last. The compound normally goes together in one or two minutes.

Most production compounds are one pass mixed, but if processing safety is marginal, accelerators (and NBC, when used), should be added at sweep-down, on the sheet-off mill, or in a second pass. Production mix cycles are short—typically 4 to 5 minutes. The compound should be dumped at or below 110°C, particularly if the accelerator is added in the mixer.

Variations on the upside-down procedure may be necessary for specific compounds. It is generally desirable to add oils and structured blacks separately (not simultaneously). If it is necessary to raise the temperature to flux resins, or if the compound mixes hot, it is advisable to add the accelerator on the sheet-off mill after the compound has cooled somewhat. Alternatively, a short second pass (about two minutes) can be made for accelerator addition as soon as the stock is cool.

On a second pass, the accelerated stock should be dumped at about 90–95°C. Dispersions of litharge and accelerators can be used to advantage, since they can be dispersed thoroughly in short mix cycles.

The sheet-off mill should be cold, and the batch should be sheeted off and cooled rapidly to minimize heat history. If possible, drop the stock through the sheet-off mill first without banding to reduce the temperature (this raises viscosity and minimizes sticking). After banding, remove stock from the sheet off mill as rapidly as possible.

Mill Mixing

Compounds of Hypalon® synthetic rubber are easily mixed on conventional mills. Because of longer heat exposure, mill mixed compounds are likely to be scorchier than those mixed in internal mixers. Full cooling water should be used on the mill except when high melting resin must be fluxed in at high temperature, in which case mixing should be finished with cooling water turned on full after the resin is dispersed. If production volume permits, resins can be masterbatched with Hypalon®, eliminating the need for excessively high mill temperatures during the normal mix operation.

Batch Size

Batch size is important in mill mixing. Correct batch weight makes possible more effective cooling and dispersion. Overloaded mills lead to excessive heat build-up and poor dispersion. Batch weights for various mill sizes can be calculated from the information shown in *Table 2*.

Mixing Procedures

A typical mill mixing time for a batch is approximately 25 minutes, depending upon the types and amounts of fillers and plasticizers. Stocks containing large amounts of plasticizers may require a much longer mixing time.

It may be necessary to start a band on a tight mill to bring about massing of the chips of Hypalon®. Since Hypalon® does not break down, no time should be spent working the polymer alone. Fillers should be

added as soon as a smooth band is formed. Following are some mixing suggestions as well as tips for achieving good dispersion:

- Magnesia and low molecular weight polyethylene should be added with the fillers. When added alone, they tend to stick to the fast roll and take the stock with them.
- Stearic acid and stearates should be added after the magnesia is incorporated.
- Stocks should not be cut while loose pigments are visible in the mix.
- Process aids should be added with the fillers whenever possible.
- Oils can be added with soft fillers and not simultaneously with reinforcing blacks.
- Resins used as softeners (e.g., coumarone-indene resins) should be added early in the mix. Many of these flux at the normal mixing temperatures.
- Add litharge and accelerators in the form of dispersions.
- Accelerators and Vanox® NBC (when used) should be added at the end of the mix. Care should be taken to flux Vanox® NBC (approximate melting point 85°C), as incomplete dispersion will result in variable heat resistance of the vulcanizates.
- If necessary to heat the mill to incorporate high melting resins, it may be more practical to add the accelerators in a second pass on the warm-up mill than it is to cool the batch while it is on the mix mill.

Table 2
Batch Weight Calculation for Various Mills

Mill Length cm (in)	Mill Roll Diameter cm	Batch Weight* Base at Sp Gr = 1 kg
76	35.6	8.5
76	40.7	10
91	41	12
107	41	17
107	46	18
122	51	23
127	46	22
152	56	38
152	61	41
183	61	53
213	61	64
213	66	70

*To calculate total batch weight, multiply numbers given above by specific gravity of compound.

For example, to mix a compound with a specific gravity of 1.5 on a 152 cm mill having 56 cm diameter rolls, multiply 38 kg \times 1.5 = 57 kg

Batch weights can vary $\pm 10\%$ from values shown depending upon type of compound and efficiency of the water cooling system.

- Stocks should not be allowed to ride the mill at any time during the mix cycle or sticking will result.
- The batch should be turned over at least once during the mix to prevent sticking. If necessary, add the fillers and plasticizer in two increments to make this possible.

Cooling and Storage

After mixing on a mill or in an internal mixer, the stock should be cooled as quickly as possible. A water dip or spray is effective, but the slabs should be *completely dry* before they are stacked. Water absorbed by compounded Hypalon® acts as an accelerator, causing an increase in the viscosity and possibly, subsequent scorching. For the same reason, mixed stocks should not be stored in conditions of *high humidity*. Mixed compounds of Hypalon® should be used as quickly as possible.

If the mixed stock is likely to be stored for more than a week before it is used, it is advisable to withhold the accelerator and add it shortly before the next processing step. Blowing agents and accelerators should always be added to sponge compounds just before use.

Stock Warm-Up

Slabs of compounded Hypalon® synthetic rubber are tough when cold and frequently will shred if fed to a tight mill. If a few slabs are passed through a more open mill until they are warmed somewhat, they will band quickly. Once a warm band is established, cold slabs can usually be fed to one end of the mill with good results. The best warmup mill conditions depend somewhat upon the subsequent operations and will be discussed under Extrusion and Calendering.

Compounds of Hypalon® should not ride the warmup mill as they tend to stick and may scorch. If the extruder or calender operation is interrupted, the stock should be cut into the pan and spread out so its heat can dissipate. It is not advisable to feed the warmup mill unless the operation is continuous and the stock is certain to be removed in a very few minutes.

Extrusion

Feed

Cold strip or pelletized stock can be used with long barrelled tuber extruders. An extruder with an undercut feed box and a rubber screw having serrated flights in the feed section is best for Hypalon® synthetic rubber.

Strip feed from a warmup mill is used in almost all hot feed applications. The stock should be warmed to a plastic state, but for each stock, experience will dictate a maximum temperature and fluidity consistent with satisfactory extrusion.

Where the short barrelled extruder is hand fed, the feed stock should be warmed up in small quantities to maintain uniform feed stock temperature. Variations in temperature cause variations in viscosity, which result in fluctuating back pressure, extrusion speed, and die swell.

Extruder Conditions

Screens and breaker plates may be used as necessary to remove contaminants, to increase back pressure, or to eliminate surging; however, they make stocks run hotter, so the number of plates or screens should be kept to a minimum. Die heads and screens should be cleaned while hot; residual stock gets tougher as it cools.

Extrusions of Hypalon® normally run best with temperatures graded upward from cool in the back end to hot in the head area; the following are typical:

	Hot Feed	Cold Feed
Screw	Cool	55°C
Feed Area	50°C	50°C
Barrel	60°C	60°C
Head & Die	95–105°C	93–107°C

With these settings, Hypalon® is fed to the screw at high viscosity, resulting in good back pressure so that air can be worked out of the stock. As the stock is worked, it moves into the warmer zones of the extruder and becomes more plastic and flows more easily. Higher die temperatures favor a smooth extrudate surface. The best extrusions are produced with dies having a streamlined flow and short lands.

The best extruder conditions depend somewhat on the compound and equipment, but normally it is best to keep temperatures as low as possible without sacrificing desired smoothness. Air free mold preps and calender feed of soft compounds of Hypalon® can be made by extrusion.

Stock Cooling

Extrusions of Hypalon® tend to be soft when hot; therefore, immediate, thorough cooling is desirable. Soap and talc are commonly used in cooling tanks. *Zinc Stearate* should not be used as noted previously.

Compounding for Extrusion

High structure blacks, such as N550 (FEF), smooth out extrusions and make them more resistant to distortion. Laminar and platey fillers improve flow characteristics. Process aids, such as low molecular weight polyethylene, or additional resins, can be used to improve flow and extrudate surface smoothness.

The compound viscosity of extrusions and hose covers of Hypalon® that are open steam cured should be compounded to a sufficiently high viscosity to eliminate flat spotting, collapse, and water spotting. The surface of stocks containing clays, silicates, and silicas are less smooth as extruded, but smoother after cure than stocks containing only whittings. Generally, blends of non-black fillers are used to get the required physicals, processability, and cure results. The fastest cure rate practical will minimize collapse and water spotting.

Compounds that are designed for lead press-curing are more tolerant of viscosity and surface smoothness as they are supported by, and cured against, the lead which controls the shape and surface of the article. Curing against lead may cause a discoloration noticeable on light colored covers. This discoloration may take place as the lead is applied, or as the result of a chemical reaction with the compound. To minimize lead press discoloration, it is desirable to minimize, or eliminate free sulfur and sulfur bearing accelerators. The presence of silica fillers and pentaerythritol will aggravate discoloration. However, many non-black compounds containing TMTD and sulfur or Tetrone® A are successfully lead press cured in production. There are many variables in production that can affect lead press discoloration which are not controlled by the polymer or compounding. Typical of these are the barrier or release coat (the type, coverage uniformity, dryness—there should be no moisture on the uncured hose as it goes into the lead), the position of the pin and die in the lead press, method of handling the billet change, etc. If lead press curing is employed, be sure to review the precautions regarding handling of lead covered in the Handling Precautions section of this bulletin.

Color pigments must be selected that are stable at the temperature of the hot lead (204–260°C). Types of pigments which have been found suitable include: phthalocyanine (blue, green), chromate yellows, iron oxides, and chrome oxide green.

Calendering

Conventional compounds based on Hypalon® 40 synthetic rubber are calendered between 60°C and 95°C. Uneven roll speeds are used and the top roll is normally maintained about 10°C hotter than the middle roll. Cool calendering helps to eliminate trapped air. However, calendering at temperatures that are too cool restricts working of the bank to eliminate trapped air, and the stock may be tough enough to carry air through the nip. Higher temperatures produce smoother surfaces, with the upper temperature limit dictated by the tendency of the hot sheet to sag, distort, or stick as it leaves the calender.

Feed

Stock should be fed to the calender at the temperature of the middle roll; cold trim should be returned to the warmup mill (not placed in the calender bank). Temperature variations in feed stock cause uneven gauge and a rough surface. Pendulum feed of strip stock is preferred, but hand feeding from the ends of the bank also gives the bank an opportunity to work air out of the stock.

Soft compounds should be fed to the calender as air free as possible. The best working bank is the smallest that will maintain a constant feed. A rolling bank is highly desirable to eliminate trapped air. The plasticity of dry compounds can be increased with 5–10 phr of a coumarone resin which melts in the range of 80–100°C. Stock should not be allowed to ride the rolls during the calender setup period, but should be moved constantly to minimize sticking.

Calendered compounds of Hypalon® should be rolled into liners. Soft stocks may have to be rewound when cooled to prevent sticking to the liners. The maximum practical calender gauge for Hypalon® is about 1 mm; soft compounds must be calendered thinner (to eliminate air) and plied up. Ply adhesion is satisfactory if at least one ply is hot and the laminate is allowed to flow somewhat as it cools in a tight liner. Ply adhesion may not be satisfactory if the plies are cooled immediately on a cold roll, before rolling into liners.

Compounding for Calendering

Release agents must be used for satisfactory calendering. The type and quantity of process aids used depend on the compound and the calendering temperatures. Too much release agent causes entrapment of air when the band folds as it re-enters the bank. Normally, the hotter a compound can be calendered, the less release agent required. Structured blacks and fillers that raise the viscosity of the compound help eliminate air and make the hot sheet easier to handle. Usually, a blend of low temperature releases (stearic acid, polyethylene glycol, and waxes) is used for low temperature calendering. Low molecular weight polyethylene is used in addition for high temperature calender release. Low molecular weight polyethylene resins are not particularly effective at temperatures below their flux points. Consult DuPont Dow Elastomers technical bulletin "Selecting a Plasticizer and Processing Aids" for more details and options on processing aids.

Curing

Molding (Press Curing)

Hypalon® synthetic rubber can be molded and cured by all the conventional methods used in industry. Mold release and color uniformity are best when chrome plated molds are used. Mold “preps” should be thoroughly dry (including any stock lubricants), or mold discoloration of non-black compounds will result. This discoloration may not be apparent until exposure to sunlight.

Mold Lubricants

The best, most consistent release is obtained with a combination of 0.5% each of Vanfre® DFL and silicone emulsions, like Dow Corning 36 emulsion, used satisfactorily in distilled water. A polyethylene emulsion, such as Lubrex® PE-40 emulsion, is also effective in some applications. Proprietary mold releases can also be used effectively in many cases.

Stock Flow in the Mold

Mold flow and release can be improved by the use of Vanfre® HYP low melt point polyethylene or resins in the compound. Since stocks of Hypalon® tend to be tough when cold, it may be desirable to preheat large mold preps (50–80°C) to speed mold closure and insure proper filling. Preheating the stock or preforms is particularly helpful in transfer molding. At molding temperatures, viscosity drops rapidly; soft or highly extended stocks may become too soft to expel trapped air.

Avoiding Defective Parts in Transfer Molding

The use of air free preps, bumping the press, the proper location of bleeders, and the adjustment of viscosity with fillers, are all useful methods of minimizing blisters and non fills. Pock marking, resulting from small amounts of air trapped by soft, hot stock, can be minimized by reshaping the preps, increasing compound viscosity, curing to a higher state-of-cure (higher modulus), or relocating bleeders. Nonfills and knit lines can be eliminated when the causes are diagnosed. Such problems may be caused by excess and “wiped” mold lubricant, incipient scorch, incompatible plasticizers or process aids in the compound, or premature curing of stock in runners and gates (in transfer or injection molds) before the cavities are filled.

Avoiding Defects in Injection Molding

Hypalon® synthetic rubber can be compounded with good strip green strength for feeding the injection molding process. The compounded strip should be coated with a clay type slab dip due to the adverse effect of zinc bearing materials on the heat resistance of Hypalon® vulcanizates.

Hypalon® processing is compatible with all current types of injection presses; ram, two stage, and in-line reciprocating single types of molds used in these presses; two plate, three plate, and hot runner or runnerless (cold runner) systems.

The Hypalon® is preheated in the barrel through a heater system and mastication by the injection screw. Recommended barrel temperatures should be in the range of 80–85°C; platen temperatures should be in the range of 166–177°C for large volume parts; and 177–190°C for small volume parts. Temperatures above 190°C should be avoided due to possible off gassing of SO₂.

Barrel residence time for the compound is controlled by plasticizer delay time (PDT). The PDT will vary from part to part depending on part volume and plasticizing speed setting. Injection dwell times should be in the range of 4–7 sec. This is required in most instances to prevent material leak back at the injection nozzle sprue when the holding pressure is released.

To cleanout, the press should be put into crash cool and barrel emptied. It is also recommended that a masterbatch purge stock be used to keep cold runner systems from premature curing. Otherwise, follow standard procedures of machine shutdown, purging, and cleanup.

The Care of Molds

Under the worst conditions, a compound of Hypalon® can give off sulfur dioxide (SO₂) which, in turn, can attack ordinary mold steel. The resultant pitting can affect the appearance of molded parts and eventually cause parts to stick and tear. Under most conditions, ordinary mold steel is not satisfactory for the extended production of molded goods of Hypalon®.

The single most effective preventive measure available to the molder is to make the molds as smooth and corrosion resistant as possible. For example, by chrome plating of ordinary steel, or using stainless steel.

The Mold—The surfaces in contact with the Hypalon® should be hard (at least 45 Rockwell C), abrasion resistant, corrosion resistant, non-porous with surface finish appropriate to the finish desired on the molded product. For extended production runs, the ideal solution is stainless steel. A less costly, second choice involves various ways of depositing chromium on the cavity walls. In the familiar electroplating method, the chromium layer should be at least 0.1 mm or 3.5 mil thick. An alternative method that has been used in the U.S. is described as electro-chemical diffusion (as sold by Armoloy). Another alternative is an iron oxide treatment (e.g., Black Magic as offered by Mitchell-Bradford).

The Compound—Hypalon® synthetic rubber contains bonded chlorine and highly reactive sulfonyl chloride crosslinking sites. High-activity magnesia, litharge, organic lead bases, Hydrotalcite, or epoxy resins are necessary, and perform two critical functions (as acid acceptors-scavengers) to neutralize acidic products which would promote polymer degradation and corrode ordinary mold steel. More information on the roll of acid acceptors can be found in DuPont Dow Elastomers technical bulletins “Types, Properties, and Uses of Hypalon®”; “Selecting A Curing System”; and “Selecting A Filler.”

Molding at lower temperatures will help reduce mold fouling and corrosion. Cure systems should be chosen to give maximum properties and cure rate at the lowest possible cure temperature. A good general purpose cure system for injection moldable Hypalon® that gives low fouling results and rapid curing is given below.

Ingredient	phr
High-activity MgO	10.00
Tetrone® A	1.25
HVA-2	.75
MBTS	1.00
Pentaerythritol	3.00

Laboratory work has also shown that the processing aid, TE-88XL (liquid), when added near the end of a banbury mix cycle, helps in mold release and reduced mold fouling.

Cleaning—Molds should be cleaned anytime they are taken out of service— even if it is just for the week-end. They should also be cleaned in production if there is evidence of any buildup. The best cleaning method is by ultrasonic vibration with caustic solution. This is less damaging to mold details than blasting.

Open Steam Curing

Autoclaves should be preheated and all steam traps should be in good working order to insure proper temperature control and eliminate condensed water causing temperature gradients.

Extrusions and rolls should be cured at high temperatures, rather than in a step cure. Rapid initiation of cure minimizes distortion, whereas slow heating aggravates it. Compound viscosity should be kept as high as practical (by polymer choice or filler adjustment) to minimize part deformation and porosity during cure. Oven curing should be avoided in the interests of best physical properties.

Vulcanizates of Hypalon® cured in open steam frequently are softer than when press cured. Compounds that contain whitening extrude with a smoother surface than compounds that contain clay and silica, but they are usually duller after open steam cure.

Continuous Curing

Hypalon® lends itself to continuous curing methods such as LCM, fluidized bed, microwave, CV, high pressure steam, and radiation curing. Hypalon®, being a polar elastomer, is responsive to microwave heating.

Curing temperatures should be held to 204°C maximum although some stocks may cure at higher temperatures without degradation.

Hypalon® can be cured by radiation (gamma and beta radiation) to give tough, usable vulcanizates. Radiation curing takes place at atmospheric pressure and does not require heating the stock. Stocks compounded for radiation curing do not require conventional sulfur bearing accelerators.

Compounding for Continuous Curing

Optimized compounds usually are designed to be high in viscosity, contain moisture scavenging desiccants, low moisture fillers and high volatility plasticizers and fast cure systems. Usually one must compound for high viscosity and green strength to minimize distortion and porosity before curing begins. The thermoplasticity of Hypalon® aggravates the green strength problem; therefore, Hypalon® 4085 or HPG-6525 will probably be necessary for zero pressure curing. The softer grades of Hypalon® are more prone to distort or sponge during the cure in the absence of external pressure. Based on production experience, a stock viscosity of 60 (ML at 100°C) or higher is desirable.

Avoid volatile plasticizers and eliminate or remove air and moisture from stock to prevent sponging. A desiccant (5–10 phr calcium oxide) should always be included to scavenge moisture, and the predispersed form is highly recommended for maximum effectiveness and uniformity. A vacuum extruder should be used to eliminate air, although it may not be necessary for stiff stocks.

It is important to select vulcanization systems that provide rapid cure rates to minimize extrudate distortion and prevent expansion of gaseous products. Consult DuPont Dow Elastomers technical bulletin “Selecting A Curing System” for more specific guidance.

Building

Developing Building Tack

The thermoplasticity of uncured Hypalon® synthetic rubber makes building operations practical, even though cold stocks normally are dry and lack surface tack. Warming is, in fact, the preferred way to “tackify” calendered sheet. Work tables heated to 50–60°C are desirable. At these temperatures, the sheet becomes pliable, and pressure (e.g., from hand rolling or wrapping) gives sufficient flow and contact between plies for knitting and ply adhesion. It is not practical to compound Hypalon® to develop a high degree of tack. If a tackifier is used, Cumar® P-10 at 10–15 phr will improve building properties.

If a solvent wipe is indicated (as in stitching down ends, seams, and splices) cyclohexanone is preferred. It gives good surface tack with little danger of causing blisters. Cyclohexanone provides a longer tack time than other solvents that might be used. Cyclohexanone is regulated as an air contaminant in the U.S. under the Occupational Safety and Health Act (refer to 29 CFR 1910.1000).

Specific Building Operations

Tank Linings

In tank lining stocks, thermal black and clay loadings are preferred for softness and pliability. Epoxy accelerated compounds are softer and more formable than similar litharge cured compounds.

Rolls

Rolls based on Hypalon® can generally be built with cold stock if tension is maintained on the sheet as the roll is built up. The preferred method of building rolls is to wind a continuous sheet of Hypalon® on the core until the desired diameter is reached. If the roll must be built of separate sheets plied up to make the required diameter, it may be necessary to preheat the stock. Rolls should be wrapped tightly with wet nylon or cotton.

Good flow and ply adhesion are obtained from the wrap pressure when the roll is cured. The ends must be tightly blocked before the roll is wrapped. Hypalon® flows before curing starts, if there is no confinement at the ends. Such flow will reduce the inward pressure exerted by the wrappings, causing porosity or poor knitting, and possibly an undersized roll.

Most rolls can be cured immediately after wrapping. Allowing an uncured roll to rest overnight may be advisable if the stock is particularly stiff and boardy. This allows flow and the development of good ply contact. If a significant amount of flow takes place, the tapes may no longer be tight and rewinding is advisable before the roll is cured. The curing of a roll based on Hypalon should be started at the full curing temperature.

Adhesion To Metal

Cements, such as Chemlok® 250 and Thixon® P-6-3, give good adhesion to metal cores when used with recommended metal preparation procedures. Epoxy cured compounds give the best adhesion to metal as well as the best formability. They will cause tendering of the wrapping tapes unless the Hypalon® is covered with a layer of another compound (i.e., SBR or Neoprene) before the tapes are applied. Adding 4 phr of calcium hydroxide to the compound can help absorb SO₂, which most commonly degrades the wrapping. Litharge cures result in poorer, but often adequate, adhesion to the cores.

Processing Hypalon® 45 and 48

Processing of Hypalon® 45 and 48 is similar to that of Hypalon® 40, but there are a few differences that should be recognized and taken into account.

At room temperature, Hypalon® 48 is tougher than Hypalon® 45 (which has crystalline properties below 50°C), and both are tougher than Hypalon® 40. However, both Hypalon® 45 and Hypalon® 48 soften rapidly as the temperature is increased, and at high processing temperatures, they have similar viscosities (see *Figure 2*). For this reason, they should be processed at higher temperatures than are the types of Hypalon® 40.

Mixing

If Hypalon® 45 or Hypalon® 48 are mill mixed, the band must be formed on hot rolls (60°C or higher). At lower temperatures, the band is rough and ragged. In fact, it may be impossible to band under the circumstances. After part of the filler and plasticizer is incorporated, full cooling should be used.

In an internal mixer, Hypalon® 45 and 48 are mixed the same as other types of Hypalon®, but because of their toughness (crystallinity for Hypalon® 45) at room temperature, a high initial mixer power draw should be anticipated (see section on Mixing.)

Warm-Up

Because of the toughness of stocks of Hypalon® 45 and 48 at room temperature, it is even more important to use the gradual warmup procedure discussed previously.

Extrusion

Extruder temperatures should be higher for Hypalon® 45 and 48 than for Hypalon® 40. Die temperatures as high as 120°C are common. The feed section should be at about 60°C, unless a lower feed temperature is needed to maintain back pressure.

Calendering

Calendering temperatures about 95°C (middle roll) should be used for plasticized Hypalon® 45 and 48. Unplasticized compounds, designed for use uncured, are calendered as hot as 150°C. At this temperature, only a small amount of process aid (e.g., 1 phr polyethylene glycol) is required. Unplasticized compounds of Hypalon® 45 should contain 0.6 phr of an anti-blocking agent, such as Kemamide® S. After thorough cooling, the film can be rolled on itself without liners (this is impractical with plasticized stocks).

Proprietary Materials

Proprietary compounding ingredients mentioned in this bulletin are listed below. This is not to infer that comparable ingredients from other sources might not be equally satisfactory.

Material	Composition	Supplier
Hypalon® Synthetic Rubber	Chlorosulfonated polyethylene	DuPont Dow Elastomers Wilmington, DE
Neoprene W Synthetic Rubber	Polychloroprene	
Tetrone® A Rubber Accelerator	dipentamethylene thiuram hexasulfide	
HVA-2 Curing Agent	N,N'-m-phenylene-dimalcicide	
A-C® Polyethylene 617A	Low molecular weight polyethylene	Allied Signal Corp. Specialty Chemicals Div. Morristown, NJ
A-C Polyethylene 1702	Low molecular weight polyethylene	
Cumar® P-10 Synthetic Resin	Coumarone-indene resin	Neville Chemical Pittsburgh, PA
Carbowax® 3350 Polyethylene Glycol	Polyethylene glycol of approx. 3350 MW	The Dow Chemical Company Midland, MI
DFDA 0053 Polyethylene	Low molecular weight polyethylene	
Chemlok® 250 Adhesive	Proprietary cement for metal/rubber bonding	Lord Corporation Erie, PA
Dow Corning® 36 emulsion	Stable emulsion of low viscosity silicone fluid	Dow Corning STI Plymouth, MI
Epolene® N-14 Polyethylene	Low molecular weight polyethylene	Eastman Chemical Prod. Kingsport, TN
Kemamide® S Fatty Amide	Stearamide	Crompton Corp. Middlebury, CT
Lubrex® PE40 Emulsion	40% emulsion of polyethylene in water	Harwick Chemical Corp. Akron, OH
High-activity MgO Magnesia	High activity magnesium oxide	Marine Magnesium Coraopolis, PA
Multiwax® 180M microcrystalline wax	Refined microcrystalline wax com- posed primarily of alkylated naphthenes and isoparaffins	Witco Corp. New York, NY
Thixon® P-6-3	Elastomers, resins and dispersed materials in solvent	Morton Intl. West Alexandria, OH
TE-88XL	Proprietary Process Aids	Technical Processing, Inc. Paterson, NJ
Vanox® NBC	Nickel dibutyl-dithiocarbamate	R. T. Vanderbilt Co. Norwalk, CT
Vanwax® H	Selected petroleum waxes	
Vanfre® DFL	Buffered phosphate ester	
Vanfre® HYP	Proprietary polymeric process aid	
Victory® Amber Wax	Microcrystalline wax	Petrolite Corp. Tulsa, OK

For further information on Hypalon® or other elastomers please contact one of the addresses below, or visit us at our website at www.dupont-dow.com

USA – Global Headquarters

DuPont Dow Elastomers L.L.C.
300 Bellevue Parkway, Suite 300
Wilmington, DE 19809
Tel. +1-800-853-5515
+1-302-792-4000
Fax +1-302-792-4450

Austria

Dolder GmbH
Brucknerstrasse 6/2/5A
A-1040 Wien
Tel. +43-1-504 21 80
Fax +43-1-504 21 93

Benelux

N.V. Sepulchre S.A.
19, av. des Nénuphars
B.P. No. 6
B-1160 Bruxelles
Belgium
Tel. +32-2-672 23 35
Fax +32-2-673 67 82

Denmark

Nordica A/S
Pilestræde 43
Postboks 2241
DK-1019 København K
Tel. +45-3315 2855
Fax +45-3315 2161

Geneva – European Headquarters

DuPont Dow Elastomers S.A.
2, chemin du Pavillon
CH-1218 Le Grand-Saconnex,
Geneva, Switzerland
Tel. +41-22-717 4000
Fax +41-22-717 4001

Finland

Oy Algot AB
Nuutisranta 15
FI-33900 Tampere
Tel. +358-3-266 1948
Fax +358-3-266 0212

France

Safic-Alcan
Département
DuPont Dow Elastomers
3, rue Bellini
F-92806 Puteaux Cedex
Tel. +33-1-46 92 64 32
Fax +33-1-46 67 04 42

Germany

DuPont Dow Elastomers GmbH
DuPont Strasse 1
D-61343 Bad Homburg
Tel. +49-6172-87 13 55
Fax +49-6172-87 13 51

Italy

Dolder-Massara
Via Cadutti Bollatesi 38/b
20021 Bollate (MI) Italy
Tel. +39-02-350081
Fax +39-02-38300725

South & Central America Headquarters

DuPont Dow Elastomers Ltda.
Alameda Itapecuru, 506 - Sala 12
Alphaville - Barueri - SP
CEP 06454-080
Brasil
Tel. +55-11-4166-8978
Fax +55-11-4166-8989

Portugal

Safic-Alcan Portugal Lda.
Rua D. Marcos da Cruz 1351
P-4455-482 Perafita
Tel. +351-22-999 82 90
Fax +351-22-996 39 27

Spain

Safic-Alcan Espana, S.A.
Division Isisa
Rocafort, 241-243
E-08029 Barcelona
Tel. +34-93-322 04 53
Fax +34-93-410 69 78

Sweden

Nordica Elastomers AB
Hamntorget 1
P.O. Box 10104
S-43422 Kungälv
Tel. +46-300-73 250
Fax +46-300-73 251

Switzerland

Dolder AG
Immengasse 9
CH-4004 Basel
Tel. +41-61-326 66 00
Fax +41-61-326 62 04

Singapore – Asia Pacific Headquarters

DuPont Dow Elastomers Pte Ltd.
1 Maritime Square #10-54
World Trade Center
Singapore 099253
Tel. +65-6275 9383
Fax +65-6275 9395

Japan

DuPont Dow Elastomers Ltd.
Dempa Bldg. 3F, 11-15
Higashi-gotanda, 1-chome
Shinagawa-ku
Tokyo, Japan 141-0022
Tel. +81-3-3444-5166
Fax +81-3-3444-6095

The information set forth herein is furnished free of charge and is based on technical data that DuPont Dow Elastomers believes to be reliable. It is intended for use by persons having technical skill, at their own discretion and risk. Handling precaution information is given with the understanding that those using it will satisfy themselves that their particular conditions of use present no health or safety hazards. Since conditions of product use and disposal are outside of our control, we make no warranties, express or implied, and assume no liability in connection with any use of this information. As with any material, evaluation of any compound under end-use conditions prior to specification is essential. Nothing herein is to be taken as a license to operate or a recommendation to infringe on any patents. While the information presented here is accurate at the time of publication, specifications can change. Please check www.dupont-dow.com for the most up-to-date information.

Caution: Do not use in medical applications involving permanent implantation in the human body. For other medical applications, discuss with your DuPont Dow Elastomers customer service representative and read Medical Caution Statement H-69237-01.

Hypalon® is a registered trademark of DuPont Dow Elastomers.

Copyright © 1998, 2000, 2002 DuPont Dow Elastomers. All rights reserved.



THIS PAGE BLANK (USPTO)